Designation: D2363 – 79 (Reapproved 2019)

Standard Test Methods for Hydroxypropyl Methylcellulose[1]

This standard is issued under the fixed designation D2363; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the testing of hydroxypropyl methylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 6
Ash (as Sulfate)	7 to 10
Chlorides (as NaCl)	11 to 14
Alkalinity (as Na_2CO_3)	15 to 18
Iron	19 to 24
Heavy Metals	25 to 29
Methoxyl Content	30 to 35
Hydroxypropoxyl Content	36 to 41
Viscosity	42 to 46
pH	47
Solids	48 to 51
Density	52 to 56

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 33.5.1.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D96 Test Method for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure) (Withdrawn 2000)[3]

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Purity of Reagents

3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

MOISTURE

4. Scope

4.1 This test method covers the determination of the volatile content of hydroxypropyl methylcellulose and, by common usage, designated moisture.

4.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

5. Procedure

5.1 Transfer 2 to 5 g of the sample weighed to the nearest 0.01 g to a tared dish (fitted with a lid) and dry for 2 h in an oven at 100 to 105°C with lid removed. Remove the dish from the oven, cover with a lid, cool in a desiccator, and weigh.

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

6. Calculation

6.1 Calculate the percent of moisture as follows:

$$\text{Moisture, \%} = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, and
B = sample used, g.

ASH—AS SULFATE

7. Scope

7.1 This test method covers the determination of the amount of residue left from igniting a sample of hydroxypropyl methylcellulose after being moistened with sulfuric acid.

7.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

8. Reagents

8.1 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

8.2 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

9. Procedure

9.1 Weigh to the nearest 0.01 g about 2 g of the sample (previously dried for ½ h at 105°C) into a tared Coors No. 1, high-form, porcelain crucible. Add 5 drops of H_2SO_4 around the inside surface of the crucible. Place the crucible inside of a loosely fitting aluminum ring (approximately 32 mm (1¼ in.) high, with 6.4-mm (¼-in.) sidewall, and 44-mm (1¾-in.) inside diameter, cut from a piece of aluminum pipe) on a hot plate. Loosely cover with a crucible cover. Carefully char the hydroxypropyl methylcellulose until all the volatiles are removed.

9.2 Cool the crucible, add 1 ml of H_2SO_4 and 2 ml of HNO_3 so that it completely wets the charred residue. Cautiously heat to dense white fumes on a hot plate. Place the uncovered crucible in a muffle furnace at 600°C and ignite until all the carbon is gone (for about 1 h). Transfer to a dessicator until cool, then weigh. (Save the residue for the Heavy Metals determination.)

10. Calculation

10.1 Calculate the percent of ash, C, as follows:

$$C = (A/B) \times 100 \quad (2)$$

where:

A = sulfated ash, g, and
B = sample used, g.

CHLORIDES—AS SODIUM CHLORIDE

11. Scope

11.1 This test method covers the determination of the total percent of chloride (bromide included if present) calculated as sodium chloride (NaCl) in hydroxypropyl methylcellulose. The sample is dispersed and the chloride titrated volumetrically with 0.100 *N* silver nitrate solution.

11.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

12. Reagents

12.1 *Ferric Alum Indicator Solution*—Add 100 g of ferric ammonium sulfate $FeNH_4(SO_4)_2 \cdot 12H_2O$ to 250 mL of water. Heat to boiling and add NHO_3 (sp gr 1.42) slowly until the red color is removed. This will usually require about 6 to 15 mL of HNO_3. Filter the solution and store in a glass bottle.

12.2 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

12.3 *Potassium Thiocyanate Standard Solution (0.1 N)*—Dissolve 10 g of potassium thiocyanate (KCNS) in 1 L of water. By means of a pipet, measure 25 mL of 0.100 *N* silver nitrate ($AgNO_3$) solution into a 400-mL beaker. Add 100 mL of water, 10 mL of HNO_3 (sp gr 1.42), and 5 mL of ferric alum indicator solution. Titrate with the KCNS solution, while stirring, until a faint persistent red color is produced. Calculate the normality, *N*, of the KCNS solution as follows:

$$N = (A/B) \times 0.1 \quad (3)$$

where:

A = 0.100 N $AgNO_3$ solution added, mL, and
B = KCNS solution required for the titration, mL.

12.4 *Silver Nitrate-Standard Solution (0.100 N)*—Grind silver nitrate ($AgNO_3$) crystals fine enough to pass through a No. 20 (850-µm) sieve and then dry for 2 h at 110°C. Prepare a 0.100 *N* solution by dissolving 16.989 g of dry $AgNO_3$ in chloride-free water and diluting to 1 L in a volumetric flask.

13. Procedure

13.1 Weigh to the nearest 0.01 g about 1.0 g of the sample (previously dried for ½ h at 100 to 105°C) and transfer to a 500-mL, wide-mouth Erlenmeyer flask. Add 250 mL of hot water and swirl for a few minutes; then cool to dissolve.

13.2 Add 5 mL of 0.100 *N* $AgNO_3$ solution and 5 mL of ferric alum indicator solution, and back-titrate with 0.1 *N* KCNS solution to the first appearance of a faint pink color.

14. Calculation

14.1 Calculate the percent of chlorides as NaCl as follows:

$$\text{Chlorides, \%} = (\left[(AB - CD) \times 0.0585\right]/E) \times 100 \quad (4)$$

where:

A = $AgNO_3$ solution added, mL,
B = normality of the $AgNO_3$ solution,
C = KCNS solution required to back-titrate the excess $AgNO_3$, mL,
D = normality of the KCNS solution, and